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METHODS OF CHEMICAL ANALYSIS OF ANTIFRICTION BEARING ALLOYS

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 7

May 1960

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ANTIFRICTION BEARING ALLOYS

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METHODS OF CHEMICAL ANALYSIS OF ANTIFRICTION BEARING ALLOYS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 9 September 1959, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The methods of analysis prescribed in this standard have been prepared with a view that they shall be useful as reference methods. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

0.3 Taking into consideration the views of the producers, consumers and testing authorities, the Sectional Committee responsible for the preparation of this standard felt that it should be related to the technological methods followed in the country in this field. Furthermore, due weightage had to be given to the need for international co-ordination among standards prevailing in different countries of the world. These considerations led the Sectional Committee to derive assistance from the 1956 Book of ASTM Methods for Chemical Analysis of Metals (published by the American Society for Testing and Materials).

0.4 This standard which is a necessary adjunct to *IS:25-1950 Specification for Antifriction Bearing Alloys (*Tentative*), requires reference to the following Indian Standard Specifications:

†IS : 264-1950 NITRIC ACID

†IS : 265-1950 HYDROCHLORIC ACID

†IS : 266-1950 SULPHURIC ACID

†IS : 1070-1957 DISTILLED WATER

0.4.1 Wherever a reference to any Indian Standard mentioned under **0.4**, or otherwise appears in this standard, it shall be taken as a reference to the latest version of the specification.

0.5 In view of the decision of the Government of India to introduce throughout the country a uniform system of weights and measures based on the metric system, all the values appearing in this standard have been expressed in the metric units only.

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*Second revision in 1966.

†Since revised.

0.6 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with *IS : 2-1949 Rules for Rounding Off Numerical Values.

1. SCOPE

1.1 This standard prescribes the methods of chemical analysis of antifriction bearing alloys having chemical composition within the following limits:

	Percent
Lead	0·10 to 80·00
Tin	4·50 ,, 93·00
Antimony	7·00 ,, 16·00
Copper	0·10 ,, 6·00
Arsenic	0·10 ,, 0·25
Bismuth	0·05 ,, 0·1
Iron	0·08 ,, 0·1
Cadmium	0·05 ,, 0·1
Zinc	0·008,, 0·01
Aluminium	0·005, *Max*

1.2 The analytical procedures appear in the following order:

	Item No.
a) *Tin* — by the Iodimetric Method	4
b) *Arsenic* — by the Distillation (Iodimetric) Method	5
c) *Antimony* — by the Potassium Bromate Method	6
d) *Lead and Copper* — by the Hydrobromic Acid Separation Method	7
e) *Copper* — by the Rapid Electrolytic Method	8
f) *Bismuth* — by the Thiourea (Photometric) Method	9
g) *Iron* — by the Photometric Method	10
h) *Cadmium* — by the Sulphide Separation (Gravimetric) Method	11
j) *Zinc* — by the Dithizone (Colorimetric) Method	12
k) *Aluminium* — by the Aluminon (Photometric) Method	13

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*Since revised.

1.3 Special Cases

1.3.1 *When zinc content* is 28 to 30 percent in a particular alloy, the method as prescribed in Appendix A shall be followed.

1.3.2 *When tin content* is 0·5 to 1·5 percent in a particular alloy, the method as prescribed in Appendix B shall be followed.

1.3.3 *When antimony content* is less than 0·5 percent in a particular alloy, the method as given under **6** shall be followed, except that the strength of standard potassium bromate solution used for titration shall be 0·005 N (*see* **6.1.8**).

2. SAMPLING

2.1 Samples shall be drawn by sawing the ingots selected in accordance with the procedure laid down in IS : 25-1950*.

2.2 Saw cuts shall be made in the ingot at points as indicated in Fig. 1 and described in the note below the figure, in such a manner as to represent the average cross section of all ingots or bars selected for each consignment or lot of metal.

2.3 No lubricant shall be used for sawing. The sawings shall be carefully gone over with a magnet to remove any particle of steel introduced while taking the samples.

2.4 The sawings, when thoroughly mixed, shall constitute the sample for chemical analysis and shall be taken as being representative of the consignment or lot.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1957†) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. DETERMINATION OF TIN BY THE IODIMETRIC METHOD

4.1 Apparatus

4.1.1 A 500-ml conical flask fitted with a three-hole rubber stopper containing an inlet tube for carbon dioxide, an air condenser, and a hole for a burette. This hole shall be kept closed by means of a glass rod until the titration is started. During reduction, a very slow stream of carbon dioxide shall be passed through the flask. When the reduction is complete, the flow shall be increased to maintain a protective cover of carbon dioxide during cooling and titration (*see* Fig. 2).

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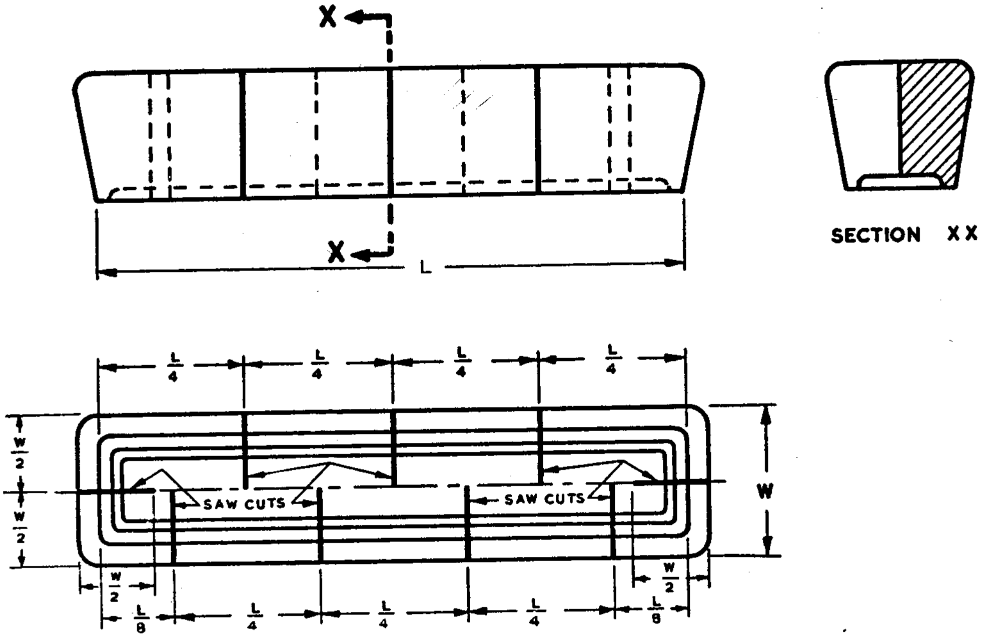
*Second revision in 1966.

†Since revised.



NOTE — With notched ingots, the saw cuts shall be distributed along in a similar manner, avoiding, if possible, saw cuts directly through a notch.

FIG. 1 METHOD OF SAMPLING OF UN-NOTCHED INGOTS

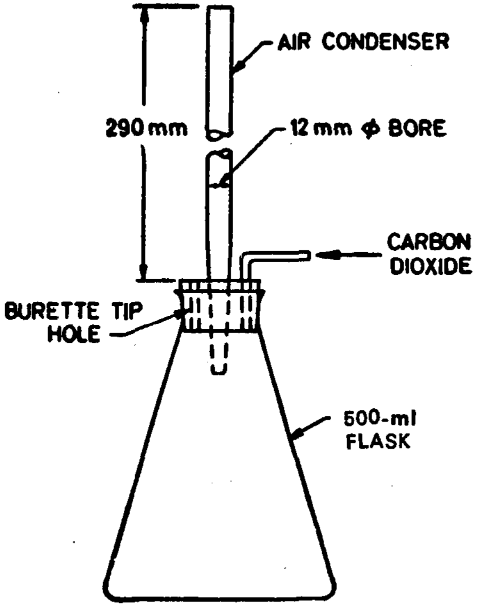


Fig. 2 Apparatus for Reduction of Tin

4.2 Reagents

4.2.1 *Concentrated Sulphuric Acid* — sp gr 1·84 (conforming to IS : 266-1950*).

4.2.2 *Potassium Bisulphate* ($KHSO_4$) — solid.

4.2.3 *Concentrated Hydrochloric Acid* — sp gr 1·16 (conforming to IS : 265-1950*).

4.2.4 *Potassium Chlorate* ($KClO_3$) — solid.

4.2.5 *Iron Wire, Strip or Rod* — of relatively pure iron containing not less than 99·85 percent of iron. The wire, strip or rod shall be properly cleaned before use.

4.2.6 *Starch Solution* — Make a suspension of one gram of soluble starch in about 10 ml of water and add it carefully to 100 ml of boiling water. Boil for two or three minutes and cool. Prepare the solution fresh as needed.

4.2.7 *Carbon Dioxide* — oxygen-free gas.

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*Since revised.

4.2.8 *Standard Potassium Iodate Solution* (KIO_3) — 0·1 N approximately. Dissolve 3·567 g of re-crystallized potassium iodate in 200 ml of water containing one gram of sodium hydroxide and 10 g of potassium iodide. When the solution is complete, dilute to one litre with water in a volumetric flask. Standardize the solution against tin or an alloy of tin, antimony, lead and copper in approximately the similar proportion as present in the sample or a similar alloy of known tin content using such an amount as to give approximately the same titration as the sample. Reduce the tin and titrate with potassium iodate solution as described in **4.3.**

4.2.9 *Standard Iodine Solution* — 0·1 N approximately. Dissolve 12·7 g of re-crystallized iodine and 40 g of potassium iodide in 25 ml of water. When the solution is complete, dilute to one litre with water and store, in a cool place, in a dark coloured glass-stoppered bottle. Standardize the iodine solution as prescribed for potassium iodate solution under **4.2.8.**

4.3 Procedure

4.3.1 Transfer 0·300 0 to 2·000 g of finely divided and thoroughly mixed sample (so as to contain 0·10 to 0·30 g of tin) to a 500-ml conical flask. Add 20 ml of concentrated sulphuric acid and 5 g of potassium bisulphate, and heat to decompose the sample. Avoid very high temperature during the initial heating; lest the sample should melt and an incomplete decomposition may result. When decomposition is complete, heat vigorously over an open flame to expel or wash down sulphur on the walls of the flask.

4.3.2 Cool and add 180 ml of water to the clear solution. Add 60 ml of concentrated hydrochloric acid and at least 5 g of iron, in the form of wire, strip or rod. Cover with a watch glass, heat to boiling and boil gently for 30 minutes. Filter off the iron, copper and antimony through a coarse filter paper or glass wool and collect the filtrate into a 500-ml conical flask and wash with hot water.. Preserve the filtrate.

4.3.3 Wash the precipitate of copper and antimony back into the original flask, add 10 ml of concentrated hydrochloric acid and a few crystals of potassium chlorate. Heat gently to effect complete dissolution of the metal. Dilute to 50 ml and boil to expel free chlorine. Add about three grams of iron and boil gently for 15 minutes. Filter through a coarse paper or glass wool into the 500-ml conical flask containing the filtrate preserved under **4.3.2**. To the combined filtrate, add 5 g of iron and fit up the apparatus as shown in Fig. 2 and heat to boiling with continuous evolution of gas for about 30 minutes. Some undissolved iron should remain after reduction. (When antimony and copper content is not appreciable, repeated reduction of tin by iron is not necessary.)

4.3.4 After the reduction is complete, cool the solution in the flask to about 10°C maintaining an atmosphere of carbon dioxide by passing the gas into the flask. Add 5 ml of starch solution and titrate with either standard potassium iodate or standard iodine solution.

4.4 Calculation

$$\text{Tin, percent} = \frac{A \times B}{C} \times 100$$

where

A = volume in ml of the standard potassium iodate or iodine solution required to titrate the sample,

B = tin equivalent of the standard potassium iodate or iodine solution in g per ml, and

C = weight in g of the sample used.

5. DETERMINATION OF ARSENIC BY THE DISTILLATION (IODIMETRIC) METHOD

5.1 Apparatus — An all-glass distillation apparatus as shown in Fig. 3 shall be used.

5.2 Reagents

5.2.1 *Concentrated Sulphuric Acid — see* **4.2.1.**

5.2.2 *Potassium Bisulphate* — solid.

5.2.3 *Carbon Dioxide* — oxygen-free gas.

5.2.4 *Concentrated Hydrochloric Acid — see* **4.2.3.**

5.2.5 *Dilute Hydrochloric Acid* — 1 : 1 (by volume).

5.2.6 *Hypophosphorus Acid Solution* (H_3PO_2) — 50 percent (*w/v*).

5.2.7 *Sodium Hydroxide Solution* — 20 percent (*w/v*).

5.2.8 *Methyl Red Indicator Solution* — Dissolve 0·1 g of methyl red in 60 ml of rectified spirit and make up to 100 ml with water.

5.2.9 *Sodium Bicarbonate* — solid.

5.2.10 *Starch Solution — see* **4.2.6.**

5.2.11 *Potassium Iodide Solution* — 10 percent (*w/v*).

5.2.12 *Standard Iodine Solution* — 0·01 N approximately. Dissolve 1·27 g of re-crystallized iodine and 4 g of potassium iodide in 25 ml of water. When the solution is complete, dilute to one litre with water and store in a dark coloured glass-stoppered bottle. Standardize against pure arsenic trioxide (AS_2O_3).

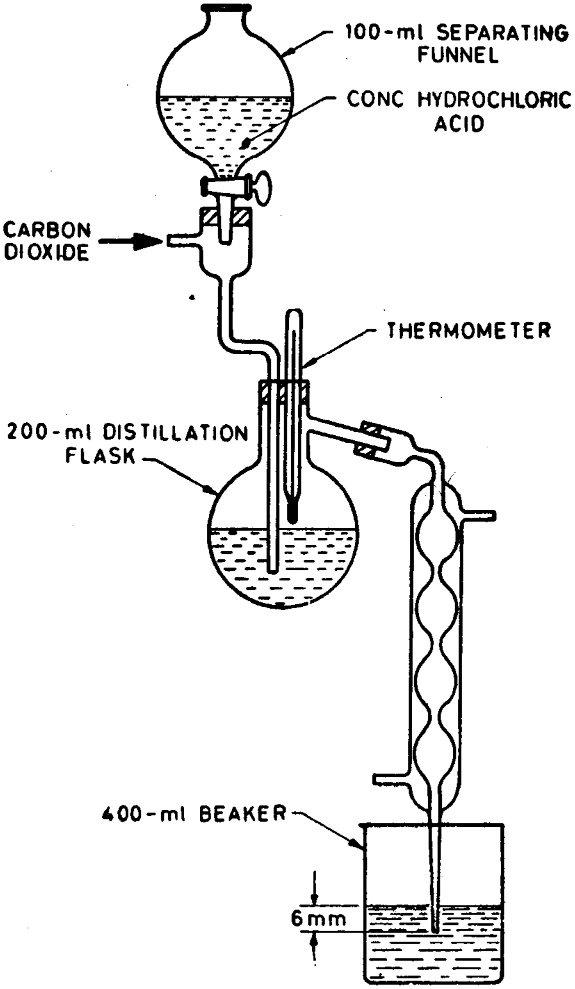

FIG. 3 APPARATUS FOR DETERMINATION OF ARSENIC BY DISTILLATION

5.3 Procedure

5.3.1 Transfer 1·00 to 5·00 g of the sample (depending on the arsenic content) to a 500-ml Erlenmeyer flask. Add 5 g of potassium bisulphate and 20 ml of concentrated sulphuric acid and heat to decompose the sample completely as described under **4.3.1.** Transfer the clear solution to the distillation flask and fit up the apparatus as shown in Fig. 3. Evaporate to white fumes while passing a current of carbon dioxide through the apparatus at a rate of 6 to 8 bubbles per second. Allow the solution to cool. Place a 00-ml beaker containing 50 ml of ice cold water under the condenser so that the tip of the condenser is dipped about 6 mm in water. Add 35 ml of concentrated hydrochloric acid and one millilitre of hypophosphorus acid to the solution in the distillation flask.

5.3.2 Place 75 ml of concentrated hydrochloric acid in the 100-ml separating funnel, and pass a stream of carbon dioxide through the apparatus. Heat the solution in the distillation flask while dropping hydrochloric acid from the separating funnel at a rate that will maintain the temperature of the flask at 110 to 112°C. Continue distillation until all the acid in the separating funnel is added.

5.3.3 Remove the receiver and rinse the end of the condenser with water. Cool the distillate in the beaker below 30°C; neutralize with sodium hydroxide solution using methyl red as indicator and add dilute hydrochloric acid solution to make it just acidic. Cool and add 8 to 10 g of sodium bicarbonate, 5 ml of starch solution and one millilitre of potassium iodide solution. Titrate with the standard iodine solution to a persistent blue tint.

5.3.4 Make a blank determination following the same procedure and using the same amounts of the reagents, but without the material.

5.4 Calculation

$$\text{Arsenic, percent} = \frac{(A-B)\,C \times 0{\cdot}037\,5}{D} \times 100$$

where

A = volume in ml of the standard iodine solution required to titrate the sample,

B = volume in ml of the standard iodine solution required for the blank,

C = normality of the standard iodine solution, and

D = weight in g of the sample used.

6. DETERMINATION OF ANTIMONY BY THE POTASSIUM BROMATE METHOD

6.1 Reagents

6.1.1 *Concentrated Sulphuric Acid — see* **4.2.1.**

6.1.2 *Potassium Bisulphate* ($KHSO_4$) — solid.

6.1.3 *Concentrated Hydrochloric Acid — see* **4.2.3.**

6.1.4 *Sodium Sulphite* (Na_2SO_3) — solid.

6.1.5 *Sodium Bisulphite* ($NaHSO_3$) — solid.

6.1.6 *Methyl Orange Indicator Solution* — 0·1 percent. Dissolve 0·1 g in 100 ml of water, filter if necessary.

6.1.7 *Standard Potassium Bromate Solution* — 0·05 N. Dissolve 1·391 7 g of re-crystallized potassium bromate in water and dilute to one litre in a volumetric flask.

6.1.8 *Standard Potassium Bromate Solution* — 0·005 N. Dilute 10 ml of standard potassium bromate solution 0·05 N (*see* **6.1.7**) to 100 ml in a volumetric flask and mix well. (This solution shall be used when antimony content of an alloy is less than 0·5 percent.)

6.2 Procedure

6.2.1 Transfer 0·500 to 5·000 g of the sample (depending on the antimony content) to a 500-ml round-bottom flask. Add 10 ml of concentrated sulphuric acid and 7 g of potassium bisulphate to the flask and carry out complete decomposition of the sample as described under **4.3.1.**

6.2.2 Cool, add 10 ml of water and shake the flask to hydrate the sulphuric acid. Add 75 ml of concentrated hydrochloric acid and one gram of sodium sulphite or sodium bisulphite. Heat the solution to 110 to 115° and boil, without cover to volatilize arsenic, until the volume has been reduced to about 60 ml. Dilute to 350 ml with boiling water and titrate the hot solution with standard potassium bromate solution, using methyl orange as indicator, which should preferably be added near the end point of the titration.

6.2.3 Make a blank determination following the same procedure and using the same amounts of the reagents, but without the material.

6.3 Calculation

$$\text{Antimony, percent} = \frac{(A-B)\,C \times 0{\cdot}0609}{D} \times 100$$

where

A = volume in ml of the standard potassium bromate solution required to titrate the sample,

B = volume in ml of the standard potassium bromate solution required to titrate the blank,

C = normality of the standard potassium bromate solution, and

D = weight in g of the sample used.

7. DETERMINATION OF LEAD AND COPPER BY THE HYDROBROMIC ACID SEPARATION METHOD

7.1 Apparatus

7.1.1 *Gooch Crucible*

7.1.2 *Platinum Electrodes* — with or without rotating stirrer.

7.1.2.1 *Cathode* — It may be formed either from a sheet or wire gauge and shall have a depositing surface of about 100 cm^2.

7.1.2.2 *Anode* — It shall be either in the form of spiral or made from wire gauge. The former is recommended when the anode serves also as a stirrer and the latter when samples contain above 0·2 percent of lead.

7.1.3 *Beaker* — It shall be 250- to 300-ml, tall-form lipless beaker with a well-fitting split cover glass.

7.2 Reagents

7.2.1 *Hydrobromic Acid-Bromine Mixture* — Add 20 ml of bromine to 180 ml of hydrobromic acid.

7.2.2 *Perchloric Acid* ($HClO_4$) — 70 percent.

7.2.3 *Hydrobromic Acid* — 48 percent.

7.2.4 *Dilute Sulphuric Acid* — 1 : 1 (by volume).

7.2.5 *Lead-Acid Solution* — Dissolve 0·5 g of lead nitrate in 200 ml of water and add with stirring 5 ml of concentrated sulphuric acid to the solution. Allow to stand for 24 hours and syphon or decant through a fine filter paper. Discard the precipitate.

7.2.6 *Concentrated Nitric Acid* — sp gr 1·42 (conforming to IS : 264-1950*).

7.2.7 *Concentrated Sulphuric Acid* — *see* **4.2.1**.

7.2.8 *Dilute Nitric Acid* — 1 : 3 (by volume).

7.2.9 *Ethyl Alcohol* (*Ethanol*) — 95 percent (by volume).

7.2.10 *Methyl Alcohol* (*Methanol*) — 95 percent (by volume).

7.3 Procedure

7.3.1 Transfer †0·1 to 2 g of the sample (depending on lead and copper content) to a 250-ml wide-mouth flask. Add 20 ml of hydrobromic acid-bromine mixture, cover and heat gently until the dissolution of the sample is complete, avoiding excessive loss of bromine. Add 10 ml of perchloric acid and heat in a well-ventilated hood while swirling over an open flame until white fumes appear. Then heat moderately and intermittently to decompose lead bromide and to expel all hydrobromic acid avoiding excessive loss of perchloric acid.

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*Since revised.

†If cadmium has to be determined from the solution reserved under **7.5.2**, take 2 to 5 g of the sample (depending on cadmium content).

7.3.2 If the solution is cloudy, showing that all the tin and antimony have not been expelled, add 5 ml of hydrobromic acid and heat again to expel hydrobromic acid and to decompose the lead bromide. Repeat the process until a fairly clear solution is obtained. Finally, add 15 ml of dilute sulphuric acid solution and evaporate to dense white fumes to expel perchloric acid. Continue heating until the volume of solution has been reduced to about two millilitres.

7.4 Determination of Lead

7.4.1 Cool, add 100 ml of lead acid solution and boil gently to dissolve the soluble salts. Cool and allow to stand at 50°C for one to two hours and finally bring to room temperature. Filter through a tared asbestos Gooch crucible and wash the precipitate well with cool lead acid solution. Reserve the filtrate for the determination of copper. Heat the precipitate, gently at first and then to constant weight at dull redness (500 to 600°C) in a muffle furnace or over a flame by suspending the crucible in a larger one so that an air space separates the crucibles.

7.4.2 *Calculation*

$$\text{Lead, percent} = \frac{A \times 0{\cdot}683\,3}{B} \times 100$$

where

A = weight in g of lead sulphate, and

B = weight in g of the sample used.

7.5 Determination of Copper

7.5.1 Dilute the filtrate, reserved under **7.4.1**, to 250 ml in the tall-form beaker and add four millilitres of concentrated nitric acid. Insert the electrodes, cover the beaker with split watch glass and electrolyse at a current density of 1 to 1·5 amperes per square decimetre. When the solution is colourless, wash down the cover glasses, electrodes and the sides of the beaker, and continue the electrolysis until deposition of the copper is complete, as indicated by failure to plate on a new surface when the level of the solution is raised.

7.5.2 When deposition of the copper is complete, with the current still on, lower the beaker slowly while washing the cathode with water. Preserve the solution in the beaker for the determination of cadmium.

Remove the cathode, rinse in water and dip the two successive baths of ethanol or methanol. Dry in an oven at 110°C for three to five minutes and weigh the deposit as metallic copper. If the deposit is dark coloured, dissolve the plate from the electrode in 15 ml of dilute nitric acid solution. Wash and remove the electrode. Heat the solution of the copper deposit to boiling to expel brown fumes, cool, and filter, if necessary. Dilute to 250 ml in tall-form beaker, add 5 ml of concentrated sulphuric acid and re-deposit the copper on the platinum cathode.

7.5.3 *Calculation*

$$\text{Copper, percent} = \frac{A}{B} \times 100$$

where

A = weight in g of copper deposited, and

B = weight in g of the sample used.

8. DETERMINATION OF COPPER BY THE RAPID ELECTROLYTIC METHOD (FOR ALLOYS CONTAINING LESS THAN 10 PERCENT OF LEAD)

8.1 Apparatus — as prescribed under **7.1.**

8.2 Reagents

8.2.1 *Hydrofluoric Acid* — 40 percent.

8.2.2 *Concentrated Nitric Acid* — *see* **7.2.6.**

8.2.3 *Ammonium Persulphate* [$(NH_4)_2S_2O_8$] — solid.

8.2.4 *Dilute Nitric Acid* — 1 : 3 (by volume).

8.2.5 *Ethyl Alcohol* (*Ethanol*) — 95 percent (by volume).

8.2.6 *Methyl Alcohol* (*Methanol*) — 95 percent (by volume).

8.2.7 *Concentrated Sulphuric Acid* — *see* **4.2.1.**

8.3 Procedure

8.3.1 Transfer one to two grams of the accurately weighed sample to a 300-ml beaker and add 25 ml of water, 10 ml of hydrofluoric acid and 20 ml of concentrated nitric acid. Warm gently to dissolve the sample. Heat to expel brown fumes and dilute to 200 ml. Add approximately 7 g of ammonium persulphate and stir to dissolve.

8.3.2 Electrolyse the solution, with agitation, at a current density of 2·5 to 3·0 amperes per square decimetre. When the solution is colourless, wash down the cover glass, electrodes and the sides of the beaker and continue the electrolysis until deposition of the copper is complete, as indicated by failure to plate on a new surface when the level of the solution is raised. When decomposition of the copper is complete, with the current still on, lower the beaker slowly, while washing the cathode with water. Remove the cathode.

8.3.3 Dissolve the plate in 15 ml of dilute nitric acid solution in a 300-ml beaker, wash and remove the cathode and heat to boiling to expel brown fumes. Add 5 ml of concentrated sulphuric acid, dilute to 200 ml and electrolyse again as prescribed under **8.3.2**. Remove the cathode, rinse it in water and dip it in two successive baths of ethanol or methanol. Dry in an oven at 110°C for 3 to 5 minutes; cool and weigh the deposit as metallic copper.

8.4 Calculation

$$\text{Copper, percent} = \frac{A}{B} \times 100$$

where

A = weight in g of copper deposited, and

B = weight in g of the sample used.

9. DETERMINATION OF BISMUTH BY THE THIOUREA (PHOTOMETRIC) METHOD

9.1 Reagents

9.1.1 *Hydrobromic Acid-Bromine Mixture — see* **7.2.1**.

9.1.2 *Perchloric Acid-Phosphoric Acid Mixture* — Mix 840 ml of perchloric acid with 160 ml of phosphoric acid.

9.1.3 *Concentrated Nitric Acid — see* **7.2.6**.

9.1.4 *Fluoboric Acid* — Pour 5 ml of hydrofluoric acid into 45 ml of saturated solution of boric acid and dilute to 500 ml.

9.1.5 *Thiourea Solution* — 8 percent (*w/v*). Dissolve 16·00 g of thiourea in 175 ml of cold water, filter and wash into 200-ml volumetric flask, dilute to the mark and mix. Prepare fresh as needed.

9.1.6 *Standard Bismuth Solution* (1 *ml* = 0·1 *mg of* Bi) — Dissolve 0·1000 g of pure bismuth metal in 10 ml of dilute nitric acid (1 : 1), add two millilitres of concentrated sulphuric acid and evaporate to fumes. Add 20 ml of water, heat to boiling and dilute to one litre.

9.2 Procedure

9.2.1 Depending on the bismuth content, transfer up to one gram of the accurately weighed sample to a 250-ml wide mouth flask. Add 10 ml of hydrobromic acid-bromine mixture. Cover and heat gently until solution of the sample is complete, avoiding excessive loss of bromine. When analysing high tin alloys, it may be necessary to add a few extra drops of bromine to ensure complete solution of the sample and oxidation of tin to the stannic state. When dissolution is complete, add 12 ml of perchloric acid-phosphoric acid mixture and heat with continuous swirling over an open flame in a well ventilated hood to expel tin and antimony bromides. When copious white fumes of perchloric acid appear, heat gently and intermittently to decompose the bromides. Avoid excessive loss of perchloric acid. Continue heating until bromides have been expelled from the flask walls and until other bromides have been converted to perchlorates. When analysing samples, where the precipitated lead bromide tenaciously resists conversion to the perchlorate, it is permissible to add a few drops of concentrated nitric acid after complete removal of stannic bromide to destroy the lead bromide. Premature addition of the nitric acid shall be avoided, since removal of most of the tin and antimony is essential. When conversion of bromides to perchlorates is complete, take to copious white fumes to expel all traces of bromine, hydrobromic acid or nitric acid.

9.2.2 Cool to room temperature, add a mixture of 40 ml of water and 5 ml of fluoboric acid solution, mix and add exactly 25 ml of freshly prepared thiourea solution. Transfer the solution to a 100-ml volumetric flask, cool to room temperature, dilute to the mark with water and shake well.

9.2.3 Transfer a suitable portion of the solution obtained in **9.2.2** to an absorption cell and record the photometric readings using a filter at 440 mμ.

9.3 Calibration Curve

9.3.1 Take a series of standard bismuth solutions containing known quantities of bismuth and a blank of 10 ml of perchloric acid-phosphoric acid mixture in separate beakers and carry out all stages of procedure as described under **9.2** and take the photometric reading for each solution using the 440-mμ filter. Construct the calibration curve by plotting photometric readings of the standard solutions against corresponding quantities of bismuth.

9.4 Calculation

9.4.1 Convert the photometric reading of the sample solution to milligrams of bismuth by means of the calibration curve and calculate the percentage of bismuth as follows:

$$\text{Bismuth, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of bismuth found in the sample solution, and
B = weight in g of the sample used.

10. DETERMINATION OF IRON BY THE PHOTOMETRIC METHOD

10.1 Reagents

10.1.1 *Hydrobromic Acid-Bromine Mixture* — *see* **7.2.1**.

10.1.2 *Perchloric Acid* ($HClO_4$) — 70 percent.

10.1.3 *Hydrobromic Acid* — 48 percent.

10.1.4 *Dilute Sulphuric Acid* — 1 : 1 (by volume).

10.1.5 *Test Lead* — lead containing not more than 0·0001 and 0·001 percent of copper and iron respectively.

10.1.6 *Sodium Acetate-Acetic Buffer Solution* — Dissolve 270 g of anhydrous sodium acetate in 50 ml of water, add 240 ml of acetic acid, cool and dilute to one litre.

10.1.7 *Hydroxylamine Hydrochloride Solution* ($NH_2OH.HCl$) — 10 g per litre. Dissolve one gram of the reagent in water and dilute to 100 ml.

10.1.8 *Ortho-Phenanthroline Solution* — two grams per litre. Dissolve 0·2 g of ortho-phenanthroline monohydrate in 90 ml of water with stirring and gentle heat. Cool and dilute to 100 ml with water.

10.1.9 *Standard Iron Solution* (*1 ml* = 0·02 *mg of* Fe) — Dissolve 0·1405 g of ferrous ammonium sulphate [$Fe(NH_4)_2(SO_4)_2, 6H_2O$] in 100 ml of dilute hydrochloric acid (1 : 19) and dilute to one litre in a volumetric flask. Prepare fresh.

10.2 Procedure

10.2.1 Transfer up to two grams of the accurately weighed sample, from which foreign iron particles have been removed, to a 250-ml wide mouth flask. Add 20 ml of hydrobromic acid-bromine mixture. Decompose the sample as described under **7.3** until clear solution is obtained. Finally, expel from the clear solution all hydrobromic acid and all but about two millilitres of perchloric acid. Add 40 ml of water depending on the copper content of the sample under analysis, add one to two grams of test lead and boil for 15 minutes. If there is any reason to suspect that the solution still contains more than two milligrams of copper, add one gram of test lead and boil for 10 minutes. Filter through a fine filter paper to remove lead, copper, etc.

10.2.2 Transfer the filtrate to a 100-ml volumetric flask. Dilute to 50 ml. Add 25 ml of sodium acetate-acetic acid buffer solution and shake well. Add two millilitres of hydroxylamine hydrochloride and 10 ml of ortho-phenanthroline solutions, dilute to the mark and shake well.

10.2.3 Transfer suitable aliquot of the solution obtained under **10.2.2** to an absorption cell and take the photometric reading of the solution using a light filter at 490 mμ.

10.3 Calibration Curve

10.3.1 Take a series of standard iron solutions and a reagent blank. Using same quantities of reagents, carry out the entire stages of procedure as described under **10.2** and record the photometric readings of all the standard solutions along with the blank one. Construct the calibration curve by plotting the photometric readings of standard solutions against the quantities of iron.

10.4 Calculation

10.4.1 Convert the photometric reading of the sample to milligrams of iron by means of the calibration curve and calculate the percentage of iron as follows:

$$\text{Iron, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of iron found in the sample or aliquot used, and

B = weight in g of the sample used.

11. DETERMINATION OF CADMIUM BY THE SULPHIDE SEPARATION (GRAVIMETRIC) METHOD

11.1 Apparatus

11.1.1 *Gooch Crucible* — A loose-bottom crucible shall be used.

11.2 Reagents

11.2.1 *Dilute Sulphuric Acid* — 1 : 4, 1 : 5 and 1 : 1 (by volume).

11.2.2 *Dilute Ammonium Hydroxide* — 1 : 1 (by volume).

11.2.3 *Hydrogen Sulphide* — gas.

11.2.4 *Ammonium Chloride* — solid.

11.2.5 *Dilute Hydrochloric Acid* — 1 : 3 (by volume).

11.3 Procedure

11.3.1 Transfer the electrolyte reserved in accordance with **7.5.2** to a 600-ml beaker and boil to expel dissolved gases. Make the solution slightly alkaline with dilute ammonium hydroxide solution and then just neutralize with dilute sulphuric acid (1 : 4). Add 25 ml of dilute sulphuric acid (1:4) in excess, dilute to 300 ml with water, heat to boiling and pass hydrogen sulphide gas into the solution for one hour. Allow it to stand until the precipitate has settled and filter off impure cadmium sulphide in a loose bottom Gooch crucible. Remove the precipitate by punching out the bottom of the Gooch crucible into a 200-ml beaker, wash and wipe the sides of the crucible with a little asbestos pulp. Add 60 ml of dilute sulphuric acid (1:5) and boil for 30 minutes. Filter, wash and add about 5 g of ammonium chloride, to the filtrate. Dilute to 300 ml with water, heat to boiling and pass hydrogen sulphide gas into the solution for one hour. Allow it to stand until the precipitate has settled and filter. Reserve the combined filtrate for the determination of zinc.

11.3.2 Dissolve the precipitate of cadmium sulphide in 25 ml of dilute hydrochloric acid solution in a weighed platinum dish, add 10 ml of dilute sulphuric acid (1 : 1) and evaporate to dense white fumes. Remove the excess sulphuric acid by heating the dish cautiously, and finally heat to 500 to 600°C. Cool and weigh as cadmium sulphate.

11.4 Calculation

$$\text{Cadmium, percent} = \frac{A \times 0{\cdot}5392}{B} \times 100$$

where

A = weight in g of cadmium sulphate, and

B = weight in g of the sample used.

12. DETERMINATION OF ZINC BY THE DITHIZONE (COLORIMETRIC) METHOD

12.1 Reagents

12.1.1 *Hydrobromic Acid-Bromine Mixture — see* **7.2.1.**

12.1.2 *Dilute Hydrochloric Acid* — 1 : 1 (by volume).

12.1.3 *Hydrogen Sulphide* — gas.

12.1.4 *Hydrogen Sulphide Wash Solution* — Saturate dilute hydrochloric acid (3:97) with hydrogen sulphide gas.

12.1.5 *Ammonium Chloride* — solid.

12.1.6 *Concentrated Ammonium Hydroxide* — sp gr 0·90.

12.1.7 *Ammonium Hydroxide Wash Solution* — Add 5 ml of concentrated ammonium hydroxide to one litre of water and dissolve two grams of ammonium chloride in it.

12.1.8 *Citric Acid* — solid.

12.1.9 *Dithizone Solution* — Dissolve 0·050 g of dithizone in two millilitres of concentrated ammonium hydroxide and 100 ml of water in a separating funnel. Extract with carbon tetrachloride, which has been distilled over anhydrous sodium sulphate, until the solvent layer is clear bright green. Discard the solvent layer which contains impurities and filter the aqueous portion through an ashless filter paper. The solution shall be prepared fresh as needed.

12.1.10 *Carbon Tetrachloride*

12.1.11 *Standard Zinc Solution* (*1 ml* = 0·000 01 *g of* Zn) — Dissolve 0·500 g of pure zinc in 10 ml of concentrated hydrochloric acid and then add 20 ml of water. Cool, transfer to a 1 000-ml volumetric flask, make up to the mark and mix well. Dilute 5 ml of this solution to 250 ml in a volumetric flask and mix well.

12.2 Procedure

12.2.1 Transfer about two grams of the accurately weighed sample to a 200-ml flask and add 20 ml of hydrobromic acid-bromine mixture. Proceed to dissolve the sample as directed under **7.3.1** and **7.3.2**. Add 100 ml of hot water to dissolve the soluble salts. Add 10 ml of dilute hydrochloric acid and cool to zero degree centigrade, filter and pass hydrogen sulphide into the filtrate for 15 minutes. Filter again and wash the precipitate with hydrogen sulphide wash solution. Discard the precipitates.

12.2.2 Boil the solution to expel hydrogen sulphide and add 5 to 6 g of ammonium chloride. Make just ammoniacal with concentrated ammonium hydroxide and boil for two to three minutes. Filter off any iron hydroxide which might separate, and wash with warm ammonium hydroxide wash solution six to seven times, collecting the filtrate and washings in a 250-ml volumetric flask. Discard the precipitate. Cool the solution, make up to the mark and mix well.

12.2.3 Pipette out a suitable aliquot of the solution (depending on the zinc content) into a separating funnel. Add 0·5 g of citric acid and adjust the *p*H to 8·0 to 9·2. Add 3 to 5 ml of dithizone solution and extract thrice with 10, 5 and 5 ml portions of carbon tetrachloride respectively. Collect the carbon tetrachloride extracts and make up to 25 ml. Prepare a standard from the standard zinc solution using the same quantities of reagents and extracting in the same manner. The quantity of standard zinc solution taken should be such that the colours match approximately. Compare the intensities of the colours in a suitable type of colorimeter.

12.3 Calculation

$$\text{Zinc, percent} = A \times \frac{H_1}{H_2} \times 0{\cdot}000\,01 \times \frac{250}{B} \times \frac{100}{C}$$

where

A = volume in ml of the standard zinc solution taken,
H_1 = height of column of standard zinc solution,
H_2 = height of column of the solution of the sample,
B = volume in ml of the aliquot taken out of 250 ml solution obtained under **12.2.2**, and
C = weight in g of the sample taken.

13. DETERMINATION OF ALUMINIUM BY THE ALUMINON (PHOTOMETRIC) METHOD

13.1 Apparatus

13.1.1 *A Mercury Cathode Cell* — The modified Melaven type mercury cathode cell with motor-driven stirrer (*see* Fig. 4), is recommended.

13.2 Reagents

13.2.1 *Hydrobromic Acid-Bromine Mixture* — *see* **7.2.1**.

13.2.2 *Dilute Sulphuric Acid* — 1 : 1 (by volume).

13.2.3 *' Aluminon ' Ammonium Aurintricarboxylate Solution* — Dissolve 0·2 g of the reagent in 100 ml of water.

13.2.4 *Glacial Acetic Acid* — 99·5 percent.

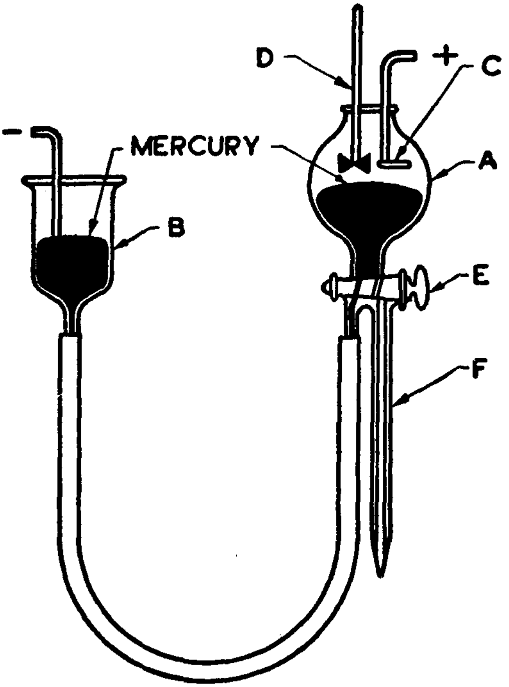
13.2.5 *Concentrated Ammonium Hydroxide* — sp gr 0·90.

13.2.6 *Concentrated Hydrochloric Acid* — *see* **4.2.3**.

13.2.7 *Standard Aluminium Solution* — (1 ml = 0·01 mg of Al). Dissolve 0·1 g of pure aluminium in 20 ml of dilute hydrochloric acid (1 : 1) and dilute to one litre in a volumetric flask. Take 10 ml from the stock solution and dilute to 100 ml in a volumetric flask.

13.3 Procedure

13.3.1 Transfer one gram of the sample to a 250-ml wide-mouth flask and add 15 ml of hydrobromic acid-bromine mixture. Cover, and heat gently till the dissolution of the sample is complete, avoiding excessive loss of bromine. If the solution is cloudy, showing that tin and antimony have not been removed, add 5 ml of hydrobromic acid and heat again to expel all hydrobromic acid. Cool and add 10 ml of dilute sulphuric acid solution. Evaporate to dense white fumes, cool, wash down the sides of the flask with water and again evaporate to dense white fumes. Dilute the solution to about 25 ml and filter off the lead sulphate.



A = Glass vessel
B = Levelling bulb
C = Platinum gauge or foil
D = A motor-driven stirrer
E = Two-way stopcock
F = Glass tubing constricted in the same manner as the burette tip

FIG. 4 MELAVEN TYPE MERCURY CATHODE CELL

13.3.2 Wash the filtrate into a mercury cathode cell containing about 50 ml of mercury and electrolyse at 3 to 5 amperes for 35 to 45 minutes while stirring the mercury and the solution. When electrolysis is complete, separate the electrolyte from the mercury, keeping a part of the current on.

13.3.3 Filter the electrolyte if it contains floating particles of loose amalgam. The volume of the solution shall now be from 40 to 75 ml. Add 5 ml of concentrated hydrochloric acid, 5 ml of glacial acetic acid and 5 ml of aluminon. Add concentrated ammonium hydroxide cautiously with constant stirring until the cloudiness disappears and the solution becomes clear, though still acidic and deeply coloured. Place a piece of litmus paper against the inner surface of the beaker and, while stirring constantly, add concentrated ammonium hydroxide at the rate of about one drop every two seconds until about two millilitres have been added, then add one drop every three to four seconds until the litmus paper turns blue. Add 5 ml of glacial acetic acid, allow the solution to stand for 10 minutes and again neutralize in the same way. Finally, add 5 ml of concentrated ammonium hydroxide and allow the solution to cool to room temperature and make the volume to 100 ml in a volumetric flask.

13.3.4 Transfer a suitable volume of the solution obtained under **13.3.3** to an absorption cell and take the photometric readings using a filter at 520 $m\mu$.

13.4 Calibration Curve — Take a series of standard aluminium solutions and a reagent blank. Using same quantities of reagents, carry through the entire procedure as described under **13.3.3** and **13.3.4** and record the photometric readings of all the standard solutions along with the blank. Construct the calibration curve by plotting the photometric readings of the solutions against the quantities of aluminium.

13.5 Calculation — Convert the photometric reading of the sample solution to milligrams of aluminium by means of the calibration curve and calculate the percentage of aluminium as follows:

$$\text{Aluminium, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = weight in mg of aluminium found in the sample solution, and

B = weight in g of the sample used.

APPENDIX A

(*Clause* 1.3.1)

DETERMINATION OF ZINC BY THE FERROCYANIDE METHOD (WHEN ZINC CONTENT IS 28 TO 30 PERCENT)

A-1. REAGENTS

A-1.1 Bromine Water — saturated solution.

A-1.2 Concentrated Ammonium Hydroxide — sp gr 0·90.

A-1.3 Ammonium Chloride Solution — Dissolve 20 g of ammonium chloride in water and dilute to one litre.

A-1.4 Ammonium Chloride — solid.

A-1.5 Dilute Sulphuric Acid — 1 : 4 (by volume).

A-1.6 Diphenylamine Indicator Solution — Dissolve one gram of the reagent in 100 ml of concentrated sulphuric acid.

A-1.7 Standard Potassium Ferrocyanide Solution — Dissolve 43·2 g of potassium ferrocyanide [$K_4Fe(CN)_6.3H_2O$] and 0·3 g of potassium ferricyanide in one litre of water and standardize against pure zinc before use.

A-2. PROCEDURE

A-2.1 Boil the *filtrate reserved under **11.3.1** to expel the dissolved gases and add excess of bromine water to oxidize hydrogen sulphide. Add concentrated ammonium hydroxide until slightly alkaline and boil it gently for a few minutes. Allow the precipitate to settle for a few minutes and filter hot. Wash the beaker and the precipitate a few times alternatively with ammonium chloride solution and water. Reject the precipitate and collect the filtrate and washings in a 500-ml flask. Concentrate the solution to 200 ml, neutralize with dilute sulphuric acid and add 15 ml in excess. Add 10 g of ammonium chloride and two drops of diphenylamine indicator solution. Heat the solution to 60°C and titrate with standard potassium ferrocyanide solution added at a moderate rate at first, but dropwise near the end point until the blue colour changes to a yellowish green. Just near the end point, dark blue solution turns suddenly yellowish green but changes back to blue. The end point is reached only when the yellowish green colour persists for 20 seconds.

NOTE — In order to get accurate results, the titration should be carried out carefully by following the procedure strictly.

A-3. CALCULATION

$$\text{Zinc, percent} = \frac{A \times B}{C} \times 100$$

where

A = volume in ml of the standard potassium ferrocyanide solution required to titrate the sample,

B = zinc equivalent of the standard potassium ferrocyanide solution in g per ml, and

C = weight in g of the sample taken.

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*The weight of the sample taken under **7.3.1** shall be such that the filtrate or its aliquot contains 0·15 to 0·20 g of zinc.

APPENDIX B

(*Clause* 1.3.2)

DETERMINATION OF TIN BY THE IODIMETRIC METHOD (WHEN TIN CONTENT IS 0·5 TO 1·5 PERCENT)

B-1. APPARATUS — (*see* **5.1**).

B-2. REAGENTS

B-2.1 Concentrated Nitric Acid — *see* **7.2.6.**

B-2.2 Dilute Nitric Acid — 2 : 98 (by volume).

B-2.3 Ammonium Persulphate — solid.

B-2.4 Concentrated Sulphuric Acid — *see* **4.2.1.**

B-2.5 Concentrated Hydrochloric Acid — *see* **4.2.3.**

B-2.6 Iron Wire, Strip or Rod — *see* **4.2.5.**

B-2.7 Dilute Hydrochloric Acid — 1 : 20 (by volume).

B-2.8 Carbon Dioxide — Oxygen-free gas.

B-2.9 Starch Solution — *see* **4.2.6.**

B-2.10 Standard Potassium Iodate Solution — 0·02 N. Transfer 20 ml of the standard potassium iodate solution, prepared in accordance with **4.2.8** to a 100-ml volumetric flask and make up to the mark with water.

B-2.11 Standard Iodine Solution — 0·02 N. Dilute 20 ml of the standard iodine solution, prepared in accordance with **4.2.9**, to 100 ml in a volumetric flask.

B-3. PROCEDURE

B-3.1 Transfer 3 to 5 g of the finally divided and thoroughly mixed sample to a 250-ml beaker and add 50 ml of concentrated nitric acid. Evaporate the solution to dryness and take up the dry mass with 25 ml of concentrated nitric acid. Dilute the solution with 100 ml of water, add a little filter paper pulp and maintain the temperature of the solution at 60 to 80°C for an hour. Filter off the residue along with the pulp and wash with hot dilute nitric acid three to four times.

B-3.2 Transfer the residue to the same beaker and add 20 ml of concentrated nitric acid, 5 g of ammonium persulphate and 20 ml concentrated sulphuric acid. Boil the solution to strong fumes. If the solution is brown, cool it, add further 5 ml of concentrated nitric acid and one to two grams of ammonium persulphate. Boil the solution carefully till it is clear (no undercomposed carbon should be left in solution).

B-3.3 Cool the solution and transfer to a 500-ml conical flask containing 100 ml of water. Add 50 ml of concentrated hydrochloric acid and about 5 g of iron, in the form of wire, strip or rod. Cover with a watch glass, heat to boiling and boil gently for 30 minutes. Cool and filter off the residue through a coarse paper or glass wool and wash twice or thrice with dilute hydrochloric acid. Collect the filtrate and washings in the 500-ml conical flask, add one gram of iron and fit up the apparatus as shown in Fig. 2. Cool the solution in the flask to about 10°C maintaining an atmosphere of carbon dioxide by passing the gas into the flask. Add few millilitres of starch solution and titrate with either standard potassium iodate or standard iodine solution.

B-3.4 Calculation — *see* **4.4.**

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